METALLIC TOOLS OBTAINED FROM CERAMIC MOULDING USING A SOL-GEL PROCESS

Teresa P. DUARTE[1], F. Jorge LINO[1], A. BARBEDO[1], J. M. FERREIRA[2]

[1]FEUP – Faculdade de Engenharia da Universidade do Porto, DEMEGI – Departamento de Engenharia Mecânica e Gestão Industrial, Rua Dr. Roberto Frias, 4200-465 Porto, Portugal,

[2]Departamento de Engenharia Cerâmica e do Vidro, CICECO, Universidade de Aveiro, 3810-193 Aveiro, Portugal

The conversion of models obtained by rapid prototyping (RP) techniques into metallic tools using a sol-gel process is presented. The process main steps are: production of a ceramic slurry which suffers a sol-gel reaction, cast the slurry into a box containing the model, stabilisation, burning, sintering and casting the metallic alloy. Processing parameters like ceramic/binder proportion, sintering time and temperatures, pouring conditions and others have a very important influence on the final quality of the metallic tools. This work presents the effect of the above parameters on the surface roughness, mechanical strength and volumetric changes from the models to the metallic tools obtained.

1. INTRODUCION

INEGI has developed and is currently refining a process to convert models obtained from RP techniques such as SL (stereolitography), LOM (laminated object manufacturing) or traditional model manufacturing techniques, into metallic tools. The aim of this process is to produce working tools by directly pouring different types of metals (aluminium, copper or other alloys) into precision ceramic moulds. These tools may be used to obtain prototypes or pre-series through different manufacturing processes.

The ceramic moulding is a precision casting process for the production of accurate castings with excellent surface finish and metallurgical integrity[1, 2].

Moulds are produced using refractory aggregates bonded with silica provided by a liquid ethyl silicate binder and are submitted to a high temperature firing treatment to produce an inert mould. The main advantages of this process are: dimensional stability, collapsibility and high resistance to thermal shock.

Ethyl silicate is a stable substance with no binding characteristics. Hydrolysis with water gives rise to monomers of silicic acid, which then polymerise in the form of an adhesive silica gel ($SiO_2 \cdot 2H_2O$), bonding the refractory aggregates. Ethyl silicate and water are immiscible, unless a mutual solvent such as ethyl alcohol is employed. This alcohol also serves to dilute the solution to the desired silica content. Hydrolysis may be carried out under either acid or alkaline conditions. However, alkaline conditions usually promote a fairly rapid gelation and consequently an acid hydrolysis is preferred (HCl). The final step of the hydrolysis reaction is:

$$(C_2H_5O)_4Si + 4\ H_2O \rightarrow SiO_2 \cdot 2H_2O + 2C_2H_5OH.$$

Different ways for promoting the gelation of an acid hydrolysed ethyl silicate can be used, however the principle of pH control seems to be the most usual method. Hydrolysed ethyl silicate solutions are usually prepared at pH values between 1.5 and 3.0. These solutions are relatively stable in this pH range and also for pH values above 7.0. Adding adequate amounts of an alkaline agent (ammonia or ammonia salts) to an acid solution increases the pH value to approximately 5.0, the binder becomes unstable and the sol-gel reaction begins. The gelation time depends on the amount of gelling agent (catalyst). The amount of catalyst is adjusted in accordance to the application[3, 4].

Many refractory materials such as zircon, alumina and aluminosilicates can be used in association with hydrolysed ethyl silicate to produce a slurry. These ceramic materials exhibit thermal and chemical stability, avoiding interaction with molten metals. The surface finish of cast tools can be improved if the refractory has a suitable particle size distribution. Careful selection of the raw materials granulometric distribution results in two particular advantages: the fine grains of the ceramics provide a smooth surface finish on the resultant casting, and the selection of a thermally stable refractory material ensures that the mould is not subject to unpredictable dimensional changes during the pre-heating and during the contact with the molten metal, thus enabling an accurate estimate of casting shrinkage[2, 5]. The secret of successful ceramic mould production lies in the material mix specification and slurry preparation. It is essential to balance the grades of refractory material with the volume of binder and the amount of gelling agent, in order to produce high quality moulds. A low binder/ceramic aggregate proportion promotes mould cracking during firing, while with a high proportion mould details can be lost and air bubbles being trapped at the pattern surface[6].

After mixing all the components (ceramic aggregate + binder + catalyst), the liquid slurry is poured into the moulding box around the pattern. Within a short period of time, controlled by the amount of gelling agent, the mould material gels to a rubbery consistency and the pattern can be separated from the mould. Following, the mould is immediately torched (stabilization) to remove alcohol and to stop the sol-gel reaction. Torching produces a very fine crazed network in the surface and inside the ceramic mould, which does not affect the casting surface, since there is no metal penetration into the fine cracks, but may improve permeability to allow the escape of air/gases during casting. The moulds are then sintered in a furnace at a temperature around 1000ºC, which ensures the elimination of combustible materials and a strong, rigid, inert, accurate and stable ceramic mould is produced. Upon heating, the silicic acid or silica gel from the binder condenses to form refractory silica cement, which provides the high strength developed during sintering[1, 2].

2. EXPERIMENTAL WORK

Different ceramic materials were mixed in different proportions, to optimise the quality of the tools obtained[6, 7, 8].

Table 1 presents the best mixture composition developed and the correspondent processing conditions.

TABLE 1
Processing conditions for the mixture developed.

Mixture Composition	60 wt% Zirconium Silicate, 30 wt% Aluminosilicates and 10 wt% Rutile
Binder	Hydrolysed Ethyl Silicate (Wacker TES 40)
Proportion Binder/Ceramic Aggregate [kg]	1 / 7.5
Mixture Time [s]/Mixture Velocity [rpm]	180 / 1850
Gelling Agent	Ammonia Hydroxide (2.5 wt%)
Room Temperature [ºC]/Humidity [%]	17-20 / 50-60
Stabilisation	Ignition immediately after demoulding

Processing parameters like amount of catalyst, sintering time and temperature and pouring conditions have a very important influence on the final quality of the metallic tools. In this communication, the effect of these parameters on the bending strength, volumetric changes and surface roughness are studied.

3. RESULTS AND DISCUSSION

The strength measured using 3 point bending tests (lots of 10 samples with 100x20x40mm) is affected by the granulometric distribution and chemical composition of the mixture and other processing parameters. Maximum strength is obtained by mixing several grading sizes, which allows the filling of the voids between larger grains, by the smaller ones. One study already published[6], indicates that the best ceramic mixture is the one presented in Fig. 1. As one can see, the developed mixture has particles from 0 to 850 µm, which are uniformly distributed in all grades.

Fig. 2 shows the effect of the amount of catalyst on the bending strength for samples sintered at 1050ºC/2h. This figure demonstrates that the amount of catalyst necessary to promote the higher bending strength is around 1.7%.

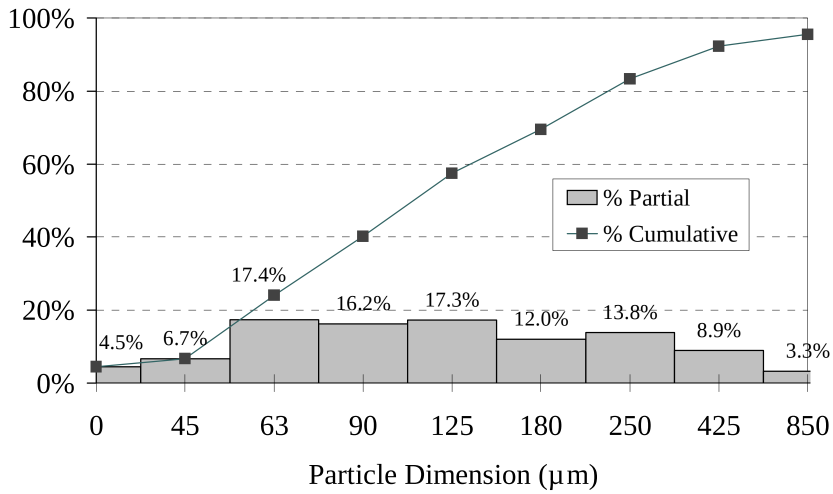

FIGURE 1
Granulometric distribution of the mixture developed.

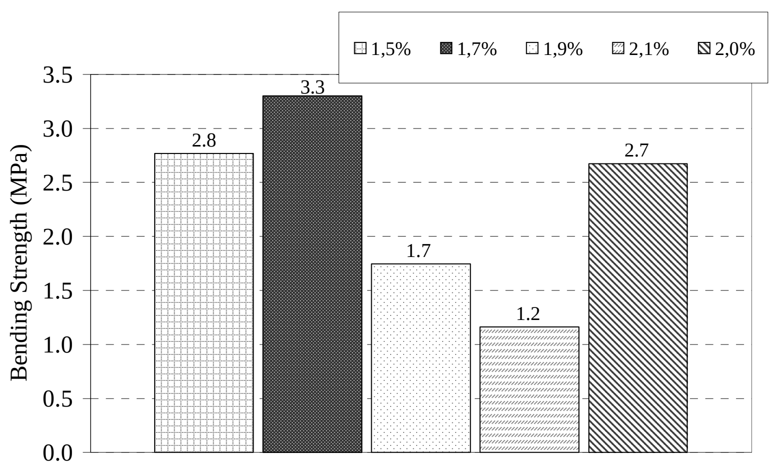

FIGURE 2
Effect of the amount of catalyst on the bending strength.

Sintering temperatures from 500 to 1500ºC and holding times from 1 to several hours are suggested in different references[2, 5]. Four temperatures and three different holding times were selected in this work as indicated in Figs. 3 and 4. These Figs. show the bending strength and volumetric changes, respectively, obtained under these sintering conditions.

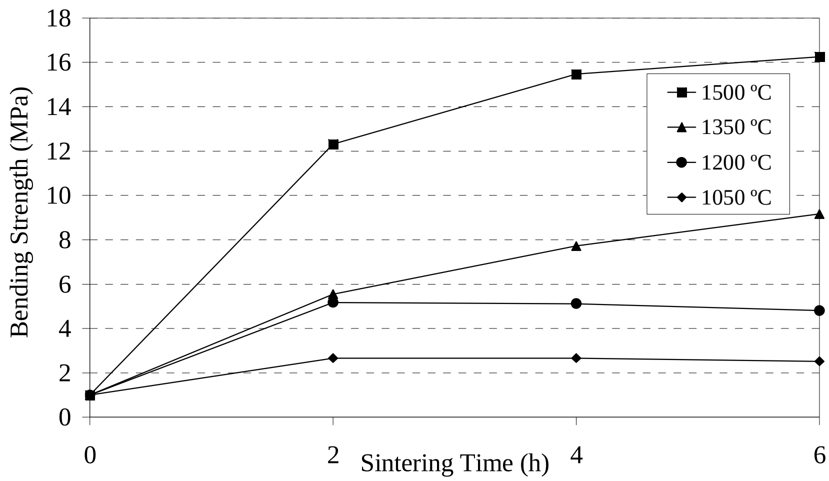

FIGURE 3
Bending strength as a function of sintering time for different temperatures.

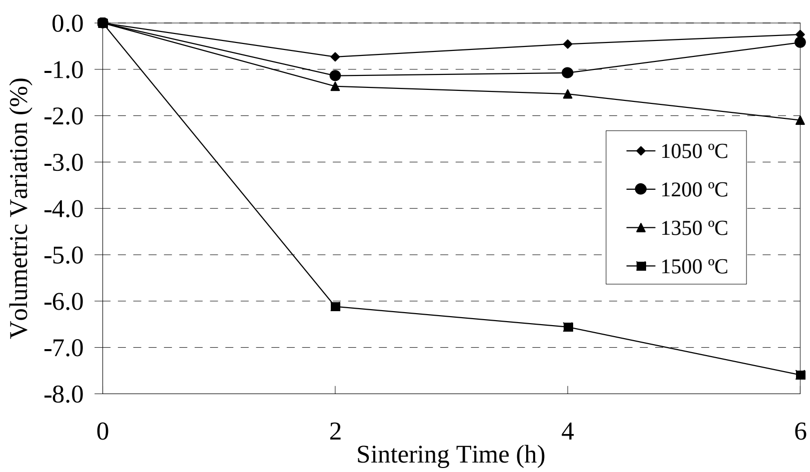

FIGURE 4
Volumetric variation as a function of sintering time for different temperatures.

Increasing the sintering temperature and time allows higher bending strengths (Fig. 3), however the shrinkage of the mould increases (Fig. 4). After holding at 1050ºC and 1200ºC for two hours, the bending strength and volumetric changes are not significant. In order to have high collapsibility and low volumetric variations (<1%) it is not recommended to sinter the ceramic moulds at temperatures above 1200ºC, for more than 2 hours.

Prototypes' surface quality has a great influence on the surface finish of the ceramic moulds. The difference between the surface roughness of the pattern and that of the final mould depends on the pouring temperature, preheating of the ceramic mould and granulometric distribution of the refractory[5]. The processing conditions employed in this study did not significantly affect the roughness value of the ceramic moulds, which was around 1.2 μm. The roughness values of the corresponding regions measured in the metallic tools obtained with different pouring conditions are indicated in Fig. 5. In this study it was found that there is no significant roughness variation (all the values of Ra are around 1.5 μm) when the pouring or preheating temperatures are varied.

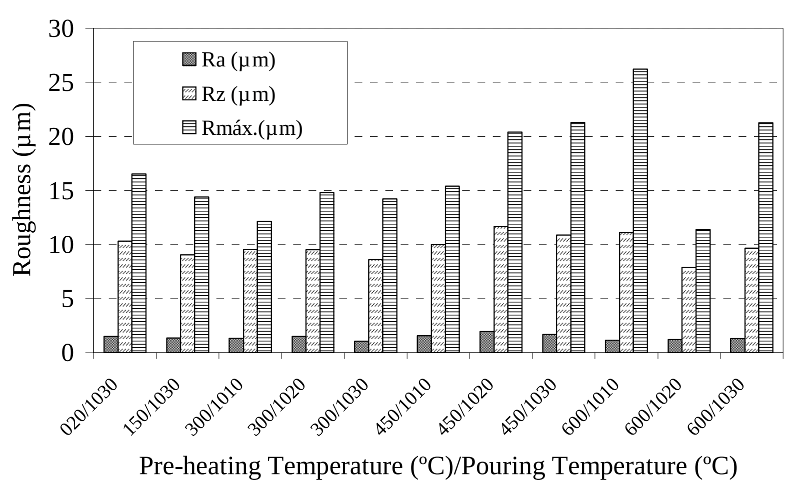

FIGURE 5

Roughness variation of the metallic tool as a function of the ceramic mould temperature (pre-heating) and the pouring temperature of a Cu-Be alloy (2.75% Be).

4. CONCLUSIONS

To produce metallic tools with tailored properties by the ceramic moulding process, it is advisable to use a mixture with ceramic particles of different sizes and uniformly distributed. This promotes a good surface quality and adequate mechanical strength to support the cast. Increasing the sintering conditions (temperature and time) increases the bending strength but the dimensional accuracy of the final metallic tool is worst due to the higher volumetric changes. The amount of catalyst also affects the bending strength with the best results being obtained with 1.7%. Casting parameters such as mould preheating and pouring temperature do not significantly affect the final surface roughness of the metallic tool.

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